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Art. II.—*A Study of the Granulation of Some Commercially Milled Victorian Flours.*

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Introduction.

When samples of flour from different flour mills in Victoria became available in 1936, it seemed an opportune time to record their granulation, especially since no general survey had been made of the granulation of wheat flour milled in Victoria. The Victorian Millowners' Association supplied the samples and a grant which made this work possible.

Wheat flour is a microscopic system containing particles of wheat endosperm ranging in diameter from $\cdot 0.1\mu$ to about 200μ. The particles are composed of starch granules of different sizes—accounting for about 75 to 90 per cent. of the total weight, a protein material called gluten—usually 8 to 20 per cent. and a small amount of cell wall. The starch granules are present in two forms—free and imbedded in the protein material. The protein is present in the aggregates of protein and starch granules. The number and size of the flour particles depend to a large extent on the milling operation.

The granulation of flour is important to both the miller and the baker. The miller is desirous of producing the flour as economically as possible, while at the same time retaining a high quality. He knows that the finer the milling products, the greater the cost of production, since more power is required for the reduction process, and fine flour requires a greater dressing or sieving surface because small particles are more difficult to dress. Quality may be affected by the heavy roller-pressure used in producing fine flours since this pressure is accompanied by elevated temperatures.

The baker judges flour to some extent by the " feel " of it and he considers the texture of the flour to be correlated with certain characteristics exhibited by the flour in the bakehouse. He is interested in the amount of water which must be used in making a dough, in the rate at which the dough develops and in the rate of gas production. It has been shown by Gründer (1935) that the fineness of a flour is related to both the rate at which water reacts with the flour when it is made into a dough and to the rate of gas production in the dough. Thus fine grinding may compensate to some extent for a low diastatic activity in the flour.

On the other hand, the same worker has shown that the elasticity and stability of the gluten are impaired by very fine grinding.

Two procedures are in general use by the miller for judging the fineness of flour. The first one is to feel the flour between the fingers. Variations between flours of very different granulation can be detected in this way but no record can be made of such variation except to describe the flour as "soft, medium, hard, smooth, gritty, &c." The "feel" of a flour is greatly influenced by the sharpness of the particles, not only by the size. Thus the final judgment is a composite of two characteristics, the shape and the size of the particles. It is obviously not possible to differentiate between them in the description of the "feel" of a flour.

The second method for judging fineness is to sift a sample of flour through a bolting cloth of known mesh size and note the proportion retained on the sieve. That this may be misleading has been shown by several workers among whom are Shollenberger and Coleman (1926). They found that flour which had been ground several times and was undoubtedly made finer by the process, sifted through a certain sized mesh less readily than did the same flour which was ground only once. Markley (1934) also found that the sieving method of estimating granulation is very inefficient. Thus neither of these methods, feeling it or sieving it, gives the miller or baker a satisfactory estimate of the granulation of flour.

Measurement of Granulation.

Methods for the estimation of the granulation of a powdered material such as flour may be divided into two groups—direct and indirect.

I. Direct Methods of Measurement.

The direct methods of measurement are of two kinds.

(*a*) *Sieve Analysis.*—Gründer (1932) pointed out that this method is only of use for particles of 60μ diameter or over. Inasmuch as the largest starch granules in Victorian flours are about 40μ in diameter and the finest mesh of bolting cloth has an opening of about 60μ, there can be no differentiation in size of starch granules by this method. In addition to the free starch granules all aggregates of starch and protein below 60μ in diameter would be free to pass through the finest mesh of silk bolting cloth.

By representing Markley's (1934) results for a commercial patent flour graphically, as in fig. 1, it is evident that up to 84 per cent. of such a flour is composed of particles having an equivalent diameter of 60μ or less and therefore a sieve having openings of 60μ diameter can differentiate only 16 per cent. of the total weight under theoretical conditions. Markley actually found, by determining the granulation of flours of varying degrees of fineness by sieving and by sedimentation, that very much less than the expected amount is passed through a sieve, as the particles passing a fine mesh are all considerably smaller than the finest mesh through which they have passed.

Gründer (1937) found that a flour sifted so as to be retained on an 150μ mesh opening and to pass a 200μ mesh, may be made up of particles of which only 20 per cent. are greater than 150μ diameter.

Several workers have contributed observations on the sifting of flour. Shollenberger and Coleman (1926) found that finely ground flour bolted more slowly than coarsely ground flour. Van der Lee (1928) considered that the rubbing of the flour on the silk sieves generated electrical forces which were partially responsible for the abnormalities encountered in sifting flour. Micka and Vrana (1930) thought that the temperature and moisture content of both the flour and air, the load on the sieves and their motion as well as the time of sifting, all affected the results of a sifting test for granulation.

(*b*) *Microscopic Measurement.*—By this method particles of angular shape are difficult to measure and small numbers of measurements are inaccurate. The method is also very time-consuming.

II. Indirect Methods of Measurement.

The indirect methods of measurement are those involving sedimentation in a gas or liquid. Sedimentation by means of an elutriator is not practical for small quantities of material.

Sedimentation in liquids may be carried out according to several modifications. They are all based on Stokes' Law of falling spheres in liquids, which is—

$$V = \frac{2}{9} g \, r^2 \frac{(D_1 - D_2)}{\eta}$$

V being the velocity of fall; g, the gravity constant; r, the radius of the sphere; D_1, the density of the sphere; D_2, the density of the liquid which is in lyophobic relation to the particle and η, the absolute viscosity of the liquid.

In any one system, the radius of the largest particle being deposited at a given time is in inverse ratio to that time since—

$$V = \frac{h}{t}$$

h, being the height of fall and t, the time of fall.

Therefore $\frac{h}{t} = \frac{2}{9} g \, r^2 \frac{(D_1 - D_2)}{\eta}$

The results are commonly represented graphically by plotting the percentage deposited against the time.

(*a*) *Odén's Method of Weighing* (1916).—This method has been applied to flour by Markley (1934). He used a mixture of carbon tetrachloride and cleaner's naptha as the liquid in which to suspend the flour particles. One pan of an automatic balance was suspended in a dilute suspension of the flour and the increase in weight of the pan noted as the particles settled out. Markley obtained his data as accumulation curves. One way of interpreting such curves is to draw tangents to the curves at successive time intervals. The points where these tangents intersect the percentage deposited axis represent the amount of material in the system with radii larger than those defined by the time points used. This method of interpretation is slow. Markley used a method of calculation from the data collected in which q represents the quantity of particles of radius greater than that defined by the corresponding time t from the percentage deposited value P.

$$q_2 - q_1 = t_2 \left(\frac{P_2 - P_1}{t_2 - t_1} - \frac{P_3 - P_2}{t_3 - t_2} \right).$$

(*b*) *Sterckx Method* (1935).—By measuring the height of the sediment accumulated in a vertical cylinder at successive intervals of time, Sterckx has developed a method for flour mill control work. It is quick and does not require special skill for its use. Unfortunately, it cannot be interpreted in terms of absolute size of particles. Also it disregards the finer particles since the height of the column of sediment is proportional to the weight, only during the early part of the sedimentation.

(*c*) *Pipette Method.*—This method is another variant of the Odén technique. Instead of weighing the sediment as it settles out, samples are pipetted off from a suspension at a known depth and time. The amount of flour in the aliquot is then determined. Gründer (1932) applied this method to wheat and rye flours. He suspended 10 grams of flour in 535 ml. of diethyl phthalate. 10 ml. samples were taken at known depths and times after the sample had been vigorously agitated.

He encountered some difficulty with the entrapped air after shaking due to the high viscosity of the diethyl phthalate. The suspended material in each pipetted sample was filtered off into a fritted glass crucible having pore openings of 5 to 10μ, washed with ether to remove the diethyl phthalate, dried at 40–50°C. and weighed. The percentage in suspension at each given time was calculated and the difference between those values and 100 is the amount that had settled out. For a given time and depth the largest particle to settle out can be calculated from Stokes' law and such values for diameters are plotted against the corresponding percentage of material settled out. To determine the amount of flour in a certain size range it is only necessary to consult the graphical representation of results.

The high viscosity of the solution used, causing entrapping of air seems at first to be the main disadvantage of this method.

(*d*) *Turbidity Measurement.*—Because the pipette method is slow, Gründer and Sauer (1937) developed a quicker method for plant eontrol work, in which the turbidity of a suspension could be observed with a photoelectric cell. This method depends on the relation between light absorption and size of particle. They found that for the range studied (about 70μ to 200μ diameter) in the system of wheat flour in diethyl phthalate, the light absorption was practieally independent of the size of particle. However, apparently measurements were not made for particles of low diameter and in fact these workers neglect the sedimentation of partieles of less than 30μ diameter. They state that about 80 per cent. of the flour is made of granules which are about 40μ in diameter.

In Australian flour it is desirable to measure a greater range of sizes than Gründer and Sauer did by means of the photoelectirc cell method.

(*e*) *Separation of Flour Constituents on Basis of Density.*—Gründer (1934) has separated wheat and rye flour into their component particles by means of mixing them with solutions having different densities. For this purpose he used varying amounts of xylol. earbon tetrachloride and dichlorethylene to obtain densities ranging from 1.42 to 1.60. By centrifuging the flour with a solution of density 1.46 to 1.48, the flour was separated into two fractions. The floating material consisted of bran particles and praetically all of the protein eontaining particles. The sediment consisted of almost pure starch with a trace of impurities such as sand, &c. These two fractions were further subdivided by repeating the centrifuging with liquid mixtures of other densities. This method of separation does not give information on the particle size but it might be used as an indication of the degree of grinding since very fine grinding will liberate more starch, which will in turn be separated by this method.

(*f*) *Surface Area by Witte's Method* (1936).—Information as to the surface area of a powder is an indication of its fineness. Witte mixed small amounts of flour with coal dust in definite proportions and noting the percentages of black and white in the mixture by means of a Leukometer, a number proportional to the surface area of the flour can be obtained The method is relatively quick to carry out but it has the disadvantage of masking any unusual mixture of fine and coarse particles such as would be revealed by a sedimentation curve.

Experimental.

In carrying out determinations of the particle size of flours milled in Victoria, a modification of the Robinson (1922) pipette method as applied to soils was used. The flour was suspended in a mixture of two parts benzene and one part of carbon tetrachloride by weight. This mixture has a fairly constant viscosity which is low enough so as not to entrap air. The viscosity and density of the solution were determined for the temperature at which the sedimentation took place. It was possible to follow the sedimentation to a point where only 5 per cent. or less remained in suspension.

25 grams of air-dry flour were placed in a thick glass bottle and 200 ml. of benzene carbon tetrachloride mixture were added. The stoppered bottles were shaken for three hours. Then the contents were poured into a cylinder $2\frac{1}{2}$ inches in diameter, having a capacity of 1,250 ml. and the volume made up to 1,200 ml. at 15°C.

20 ml. aliquots were withdrawn at varying times and depths after the suspension had been thoroughly mixed. The aliquots were placed in small tared florence flasks, the liquid distilled off on a water bath and the flasks and contents dried and weighed. Sedimentations were carried out in duplicate in a constant temperature cupboard at 15°C.

The average density of the finest and coarsest samples of flour was determined by placing a known amount of the flour in a pycnometer and filling the pycnometer with the benzene-carbon tetrachloride mixture of known density. The volume of the flour could then be determined and its density calculated. The average density was found to be 1.476 ± 0.006.

Knowing the average density of the flour particles and the density and viscosity of the liquid, by means of Stokes' law, the equivalent radii of the flour particles were calculated corresponding to different falling velocities.

From the percentage in suspension, the percentage deposited may be calculated and plotted against the corresponding equivalent radius of particle. The curves No. 55, 31, 27, and 58 in fig. 1 were obtained in this way.

Knowing the radii and having summation sedimentation curves as in fig. 1 for each sample, the surface area exhibited by 1 gram of each flour may be calculated. In Table I. the surface area of 1 gram of flour is tabulated for each flour examined. Instead of recording the surface area as square centimeters per gram of flour, one may construct summation curves (fig. 2) based on surface area instead of on weight. Such a representation emphasizes the extraordinary influence which the small granules have on the total surface area.

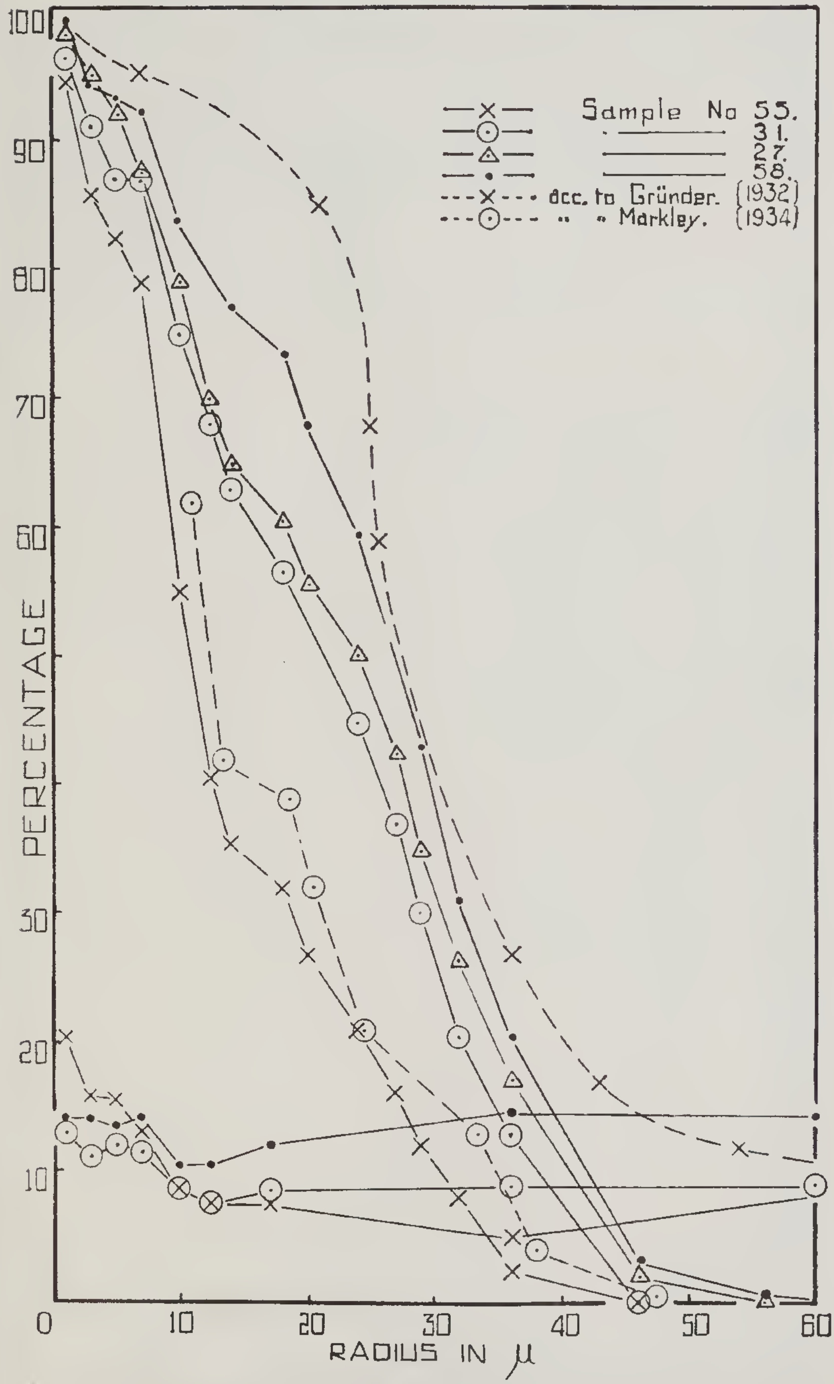

FIG. 1.—Summation curves illustrating the granulation of six samples of flour and the corresponding protein content of different size fractions for three of the samples.

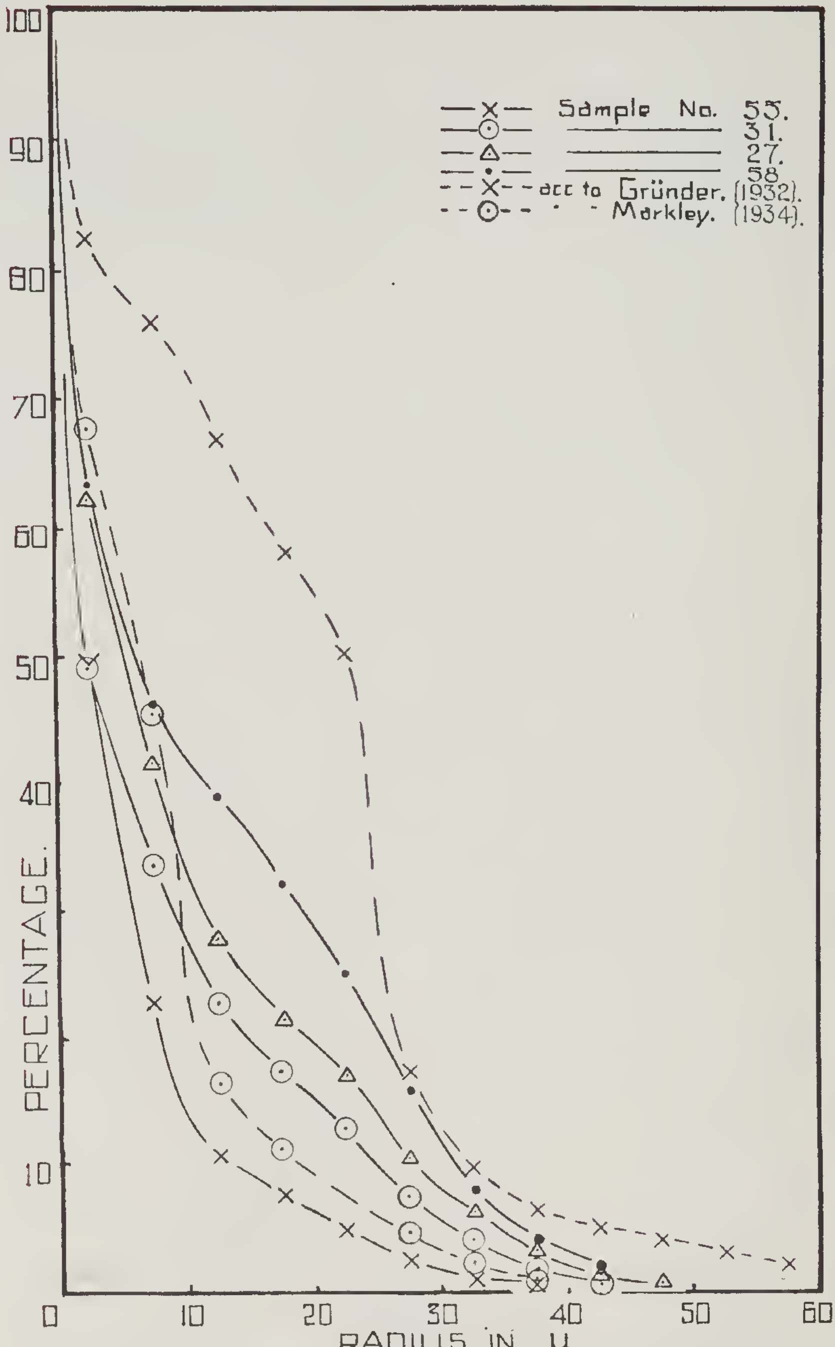

FIG. 2.—Summation curves illustrating the percentage distribution of the surface area in relation to the radius of particle for six samples of flour.

The nitrogen content of the 20 ml. aliquots was determined by the Kjeldahl method for three flours. The protein values (N x 5.7) obtained for a finely ground flour of low protein content (No. 55), a coarsely ground flour of high protein content (No. 58), and a moderately coarse flour of low protein content (No. 31) are shown together with their granulation curves in fig. 1.

A description of the flour as to the constituent wheats used, protein content, surface area per gram, and the percentage by weight above 20μ radius are given in Table I.

Discussion.

Fig. 1 gives a series of six summation curves. Three of them are for Victorian flours, one for a Canadian flour, and two from the literature for comparison. The three curves given for the Victorian flours represent the finest (No. 55) and coarsest (No. 27) flours in the collection of twenty-three samples as well as one (No. 31) of moderately coarse granulation. The Canadian flour (No. 58) was milled in Canada from Manitoba hard wheat and was imported into Australia for use in comparative baking tests. One curve represents graphically the results as published by Markley (1934) for a patent flour of commercial origin milled in America from hard spring wheat, and the remaining curve, the granulation of a wheat flour of usual commercial superfine quality in Germany according to Gründer (1932).

It is readily seen that all the flours from Victoria are finer than the Canadian flour represented in fig. 1, but the sample examined by Markley lies within the range covered by the Victorian samples. The interpretation of the curves as to size of particle is very dependent on the specific gravity of the flour. Markley does not indicate what value he used for the density of the flour. Gründer does give this value as 1.4000 in one paper (1932) and as 1.458 in another (1935). In still another place (1934) he indicates a method of separation of flour into two portions with a solution whose specific gravity was 1.46 to 1.48, such that 64 per cent. of the flour is in the floating portion and 36 per cent. in the heavier fraction, the latter being mainly free starch granules.

It is to be expected that Gründer's results will differ from those obtained in this investigation, since he was working with flours milled under different conditions and he was using a different density value for the flour with which he worked. The main difference between the granulation curve for a commercial flour obtained by Gründer (1932) and that obtained by Markley (1934), as well as those obtained in this investigation, lies in that portion of the curve for the particles of low radii, i.e., below

30μ radius. An explanation of the probable reasons for the difference in the two types of granulation curves will be given elsewhere.

Taking the percentage with radius above 20μ as an indication of the relative coarseness of the flour (see Table I.), 65 per cent. of the Victorian samples are made up of particles in which 40 per cent. or less are above 20μ radius. The remaining 35 per cent. of these Victorian flours contain 40 to 55 per cent. of their total weight as particles of radius greater than 20μ. 68 per cent. of the Canadian sample is made up of particles of greater than 20μ radius.

The percentage of protein in the suspended material rises slightly as the sedimentation progresses with the finely ground flour, but it remains more nearly constant in the coarsely ground flour (see fig. 1). This alteration in protein content of the suspended material indicates that fine grinding allows of the liberation of more of the starch from the protein material, at the same time particles of higher than average protein content are freed, and these remain in suspension longer because of their slightly lower density. It is evident that the use of an average value for density of the flour particles over the whole range of sedimentation will not lead to very serious error in interpretation of the sedimentation curves.

The curve showing the relation between the equivalent radius and the summation of the surface area of the particles (fig. 2) is a better indication of the fineness of a flour than the curve showing the summation weight or sedimentation curve in relation to the radii of the particles as in fig. 1. For certain purposes such as comparing the fineness of different flours, the surface area of 1 gram of flour in square centimetres is very useful. Such values from Table I. for the 22 samples of Victorian flour were used in compiling the frequency data in Table II. The values for surface area per gram of flour give a normal frequency distribution.

Theoretically, in forming a dough by addition of water, intimate contact between flour particles and water should be achieved in less time in a finely ground flour than in a coarsely ground one. Likewise, the contact between added dough improvers, yeast, &c., and the flour would be more readily established in finely ground flour. Gründer (1935) has furnished experimental proof of the correlation between fineness of a flour and both the dough developing time and the rate at which carbon dioxide is generated in the dough. By the use of the Brabender fermentograph and farinograph apparatus he was able to show that as the surface area of a given flour increased, so the carbon dioxide produced increased and the dough developing time shortened. In controlling the quality and uniformity of his product, this is of direct use to the flour miller. The control of granulation may be achieved either by mixing flours of known granulation or by controlling the grinding and dressing of the flour from a given blend of wheats.

TABLE I.

Sample No.	Mill.	Percentage above 20μ radius.	Surface area in cm^2 per gram of Flour.	Percentage Protein on dry basis.	Description of Sample.
58	N	68·0	1,504	14·1	Manitoba hard wheat
27	C_2	55·6	1,705	16·3	"Strong" flour
34	H	53·0	1,949	8·9	"Dundee" and "Baringa" wheat from South Riverina, N.S.W., and "Ghurka," "Ranee," and "Waratah" from Rutherglen and Wangaratta
23	B_1	48·0	2,023	8·5	"Ghurka" from Rupanyup District
31	E	52·0	2,065	9·1	"Dundee" wheat from Charlton District
22	B_2	41·0	2,135	11·4	Blend of Mallee wheats
32	F	47·0	2,147	9·4	"Ford," "Baringa," and "Dundee" wheat from N.S.W. (41 per cent.); 1934–35 wheat from Waaia District (mixed) (17 per cent.); 1935–36 wheat from Katamatite and Waaia Districts (mixed) (42 per cent.)
28	D	41·5	2,148	9·7	Wheat from Riverina and Victorian Wimmera Districts
29	D	41·5	2,230	9·7	" " " " "
26	C_2	39·5	2,256	11·3	"Medium" strength flour
36	J	39·0	2,284	10·8	"Ranee 4H" wheat with small amounts of "Free Gallipoli" and others. Wheat from 3 seasons—⅓ 1933–34, Millewa District; ⅓ 1934–35, Walpeup District, and ⅓ 1935–36, Millewa District
24	C_1	40·0	2,292	11·1	
25	C_2	37·0	2,300	9·6	"Weak" flour
35	I	39·0	2,313	9·9	Mainly "Ranee" and "Ghurka" with mixture of premium wheats (no Gallipoli used) from Northern and Mallee Districts
30	E	35·0	2,350	10·0	50 per cent. from Charlton District, 30 per cent. from Stations north of Charlton, and 20 per cent. from Stations on Ultima Line
38	L	36·8	2,358	9·8	
21	B_1	38·0	2,359	12·5	Blend of specially selected "strong" North-west Mallee and Riverina wheats
37	K	37·0	2,363	10·8	
20	B_1	35·5	2,367	11·1	Selected Victorian wheat of f.a.q., quality
33	G	30·5	2,567	9·7	Mixed varieties grown in Wycheproof, Murrayville and Donald Districts
54	C_1	32·5	2,590	10·3	"Strong" flour
53	C_1	29.0	2,682	12·4	"Extra strong" flour
55	C_1	26·7	2,784	8·7	"Weak" flour
	Mean of Victorian Samples ..	39·7± 9·7	2,284± 242	10·5± 1·6	

Of the samples of Victorian flour examined, the surface area per gram of the finest sample (No. 55) is 63 per cent. greater than that of the coarsest sample (No. 27). These two samples differ widely in their protein content, the coarsest sample being higher. In contrast, two samples (No. 55 and 23) having an almost identical protein content differ in their granulation such that the finer (No. 55) has a surface area 35 per cent. greater than that of the coarser sample. In considering all the samples examined there is a significant correlation of —0.538 between protein content and granulation.

TABLE II.

Area in sq. cm. per gram of Flour.	Frequency.
1,700–1,899	1
1,900–2,099	3
2,100–2,299	7
2,300–2,499	7
2,500–2,699	3
2,700–2,999	1

With differences in surface area per gram of the magnitude indicated in Table I., one might expect that such differences would be reflected in the bakery and no doubt those differences which do exist are partially bound up in the flour granulation.

For wheat of a given quality for a given purpose, it seems probable that there is an optimum granulation, just as there is an optimum diastatic capacity, protein content, &c. As yet very little is known about connecting the granulation of a flour with its properties except for the work of Gründer on flour milled in Germany.

It is to be expected that wheat of different varieties, grown in different places, may yield a flour of varying granulation when milled in different flour mills. If, however, the wheats from various sources were milled under uniform, set conditions, the granulation of the flour might be an indication of certain characteristics of the wheat from which it is made. Cutler and Brinson (1935) have found that by grinding whole wheat under uniform conditions, the resulting granulation is an indication of the class to which the wheat belongs, soft, medium, or hard, and the granulation number determined by them is correlated with the percentage of starchiness—a high proportion of fine particles being positively correlated with a high percentage of starchiness.

Further work carried out by Fifield (1934) of the United States of America Department of Agriculture indicates that the granulation number is correlated with the protein content of the grain and to a certain extent with the locality where it was grown.

Now the grinding of whole wheat meal is a different process from the grinding of wheat into white flour, but if there are inherent properties in the wheat kernel which affect the granulation number, one would expect that there would be some evidence of those properties exhibited in the grinding of wheat into flour.

If the setting of the rollers in the flour mills and the dressing processes were similar in the different mills, the flour produced might be expected to reflect that uniformity in production by varying in granulation according to the wheat used. As the granulation of the flours examined does not reflect such an indication of quality as the protein content, one can only conclude that the granulation of the commercial flours examined is almost solely the result of the varying opinions of the flour millers as to what constitutes a desirable granulation superimposed on the inherent properties of the particular blend of wheat which they have at hand for milling.

One must also infer that it is very unwise to draw any conclusions as to the quality of a flour from the "feel" of it for the granulation of these commercial flours in Victoria show no relation to one of the common measures of quality, namely the protein content.

It is of interest to note that certain Victorian flour mills tend to produce a flour of fairly uniform granulation irrespective of the protein content of the flour. This is true of samples from mills B_1, B_2, and D. (Table I.).

Other mills, E and C, produce flours of varying granulation and with different protein content, there being no relation between the protein content and the granulation of the products of the individual mill.

Since this paper was read the authors have learned of the work on Flour Granularity carried out by D. W. Kent-Jones, E. G. Richardson and R. C. Spalding, which was reported in the *Journal of the Society of Chemical Industry*, Vol. 58, pp. 261-267, August, 1939.

Summary.

The difficulties and inaccuracies encountered in estimating the granulation of flour particles by feeling the flour between the fingers and by sieving have been enumerated.

Other methods for the measurement of flour granulation have been discussed. The sedimentation method used in this investigation has been described and the results obtained when it was applied to 22 samples of commercial Victorian flour and one of Canadian flour are described.

It has been pointed out that there is a low but significant correlation in these commercial flours between quality as revealed by their protein content and granulation.

Because of the differences in both the blending of the original wheats and in the milling practice followed by the various millers, it is to be expected that the granulation of the samples will not be closely related to such a quality estimate as that furnished by protein.

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